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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification <sup>6</sup> : A23D 9/007, 9/04, A23L 1/221, 1/36, 1/39 A1</p>	<p>(11) International Publication Number: <b>WO 98/47386</b> (43) International Publication Date: 29 October 1998 (29.10.98)</p>
<p>(21) International Application Number: PCT/EP98/02269 (22) International Filing Date: 15 April 1998 (15.04.98) (30) Priority Data: 97201188.6 24 April 1997 (24.04.97) EP (34) Countries for which the regional or international application was filed: NL et al. (71) Applicant (for all designated States except AU BB CA CY GB GH IE KE LK LS MN MW NZ SD SG SZ TT UG): UNILEVER N.V. [NL/NL], Weena 455, NL-3013 AL Rotterdam (NL). (71) Applicant (for AU BB CA CY GB GH IE KE LK LS MN MW NZ SD SG SZ TT UG only): UNILEVER PLC [GB/GB], Unilever House, Blackfriars, London EC4P 4BQ (GB). (72) Inventors: GUBLER, Gregor; Lipton-Sais, Grafenau 12, CH-6301 Zug (CH). MACMILLAN, Joanna, M., S.; Lipton-Sais, Grafenau 12, CH-6301 Zug (CH). VAN OOSTEN, Cornelis, Willem; Unilever Research Vlaardingen, Olivier van Noortlaan 120, NL-3133 AT Vlaardingen (NL).</p>	<p>(74) Common Representative: UNILEVER N.V.; Patent Dept., P.O. Box 137, NL-3130 AC Vlaardingen (NL). (81) Designated States: AL, AM, AT, AU, AZ, BA, BE, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TO). <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>
<p>(54) Title: POURABLE FAT COMPOSITIONS CONTAINING A THICKENER (57) Abstract Pourable fat compositions, provided with thickeners having a good closed-shelf life are obtained, if the compositions have the following composition: 0-45 wt.% herbs, spices, nuts or seeds; 5-60 wt. % of thickener; 0-5 wt.% of emulsifier; 0-10 wt.% of milk proteins; 0-10 wt.% of egg yolk powder; 1-10 wt.% salt; 50-89 wt.% of fat blend, comprising: (i) 90-99.8 wt.% liquid oil; (ii) 10-0.2 wt.% hard fat, that can form crystal network; 0-20 wt.% of flavour-compositions.</p>	

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## POURABLE FAT COMPOSITIONS CONTAINING A THICKENER

Fat-compositions that contain herbs and/or spices are known in the prior art (c.g. US 4,572,836). However so far these  
5 compositions are only known as pastes, or other non-pourable herbs-dispersions, wherein the fats applied in the paste have high trans-contents; these products contain high levels (>12%) of cooking salt and other inorganic or organic salts for microbiological keepability and taste  
10 boosting effect reasons.

Therefore no solution existed for a long felt need, i.e. a fat product, that could be handled easily, e.g. by being in a pourable form, which contained high concentrations of herbs, spices, nuts, cheese and/or seeds and low  
15 concentrations of the unhealthy trans-fats, while its closed shelf life was at least 6 months.

In EP 21 483 it is disclosed that flavor enhancing materials in the form of ionic salts, such as sodium  
20 chloride can be incorporated in liquefiable fats if they are first micronised to a particle size of 1-10 microns. In that instance compositions can be obtained, which are stable for more than 2 hours. Incorporation of a hardstock fat can increase the stability to about 6 months. Examples  
25 of hardstock fats mentioned are fully hydrogenated normally solid fatty triglycerides with an IV <15. These hardstocks can contain fatty acid residues with 12-22 C-atoms. Examples of hardened oils are given on p.8, 1.3-8, however this listing does not include hardened rape seed oils.  
30 Although the disclosure of this document is very generic in the definition of the flavor enhancing component, it is also clear from this document that the only flavor

enhancing components, that were actually considered, are ionic salts. Therefore this document does not provide directions how to come to pourable fat compositions with very high contents of herbs and/or spices, that would be stable for long periods of time (= having long closed shelf life).

From WO 94/08470 salad dressings are known, wherein the fat phase can contain a hardened rapeseed oil component. The dressing further can contain up to 4 wt% of a herb or spices. As this reference relates to water-containing dressings, this document does not provide a solution for systems wherein no waterphase is present and that contain high amounts of herbs and/or spices.

15

In our co-pending European patent application 96202913.8 we disclosed a fat-composition that fulfils all above requirements. So accordingly above EP patent application concerns a pourable fat-composition, that is ambient-stable, and wherein the fat-composition comprises:

20

10 - 45 wt % of herbs, spices, nuts and/or seeds

0 - 20 wt % of cheese particles

1 - 10 wt % of an edible salt

50 - 89 wt % of a fat blend, comprising:

25

(i) 90 - 99.8 wt % of liquid oil

(ii) 10 - 0.2 wt % of a hard fat component, that has the ability to form a crystal network in the endproduct, preferably being hardened high erucic rapeseed oil, while the total-composition is flavoured by the presence of 0.5 - 5 wt % on total composition of flavour-ingredients.

30

Above compositions are substantially free of trans fatty acids. In fact the amounts of trans acids are less than 5 wt%, preferably less than 0.5 wt%. (Calculated on total fatty acid content.)

5

Although above compositions can be applied successfully in many application these compositions are unsuitable for application in products like Ready-to-use Roux; Ready-to-use brown or white sauces or Ready-to-use Sauce  
10 Hollandaise, optionally obtainable by adding an aqueous liquid and applying heat. In order to make them suitable for those applications it was found that a thickener should be incorporated in the compositions according to EP 96202913, while the amount of spices, herbs, nut and/or  
15 seeds could be lowered or could even be absent.

The novel products according to the invention therefore comprise pourable fat compositions, that are ambient-stable, wherein the fat-composition comprises:

- 20 0 - 45 wt % of herbs, spices, nuts and/or seeds
- 5 - 60 wt % of a thickener, in particular selected from natural or modified starches, natural or modified gum
- 0 - 10 wt % of milk proteins
- 0 - 10 wt % of egg yolk powder
- 25 0 - 5 wt % of emulsifier
- 0 - 35 wt % of meat-extract or gravy flavour
- 1 - 10 wt % of an edible salt
- 30- 89 wt % of a fat blend, comprising:
  - (i) 90 - 99.8 wt % of liquid oil
  - 30 (ii) 10 - 0.2 wt % of a hard fat component, that has the ability to form a crystal network in the

endproduct, preferably being hardened high erucic rapeseed oil,

while the total-composition is flavoured by the presence of 0 - 20 wt % on total composition of flavour-ingredients.

5 The thickener is suitably selected from natural or modified starches, such as natural wheat flour, modified starch e.g. Col Flo 67® (from National Starch), natural or modified gums, such as carrageenan gum, guar gum, xanthan gum, locust bean gum etc. Gelatin from any source can also be  
10 used as thickener.

The milk-proteins can be added as skimmed milk powder, but also as full milk powder. The amount used preferably ranges from 1-8 wt%, in particular from 2-6 wt%.

15

The egg-yolk powder can be dried natural egg-yolk, but can also be an enzymically modified egg-yolk powder, such as (spray)dried egg-yolk powder stabilised using an enzyme having phospholipase A2 activity.

20

The meat-extract or the gravy flavour is typically present in the brown sauces. Typical amounts being 2-28 wt%.

The composition can be stabilised by addition of low  
25 amounts of preservatives, such as 0.05-2 wt % of sorbic acid or salts hereof.

The products as disclosed above can be mixed up with hot water, hot milk, stock, or other hot aqueous liquid, i.e.  
30 having a temperature of >80°C, preferably 85-99°C, in particular 90-95°C, without having lumping or caking problems. The amount of water, milk, or aqueous liquid that

can be used depends on the thickness and taste requirements of the user, but in fact no limitations exist hereon. Typical dilution rates are 1 part of the composition as described above and 3 to 8 part, preferably 4-6 parts of aqueous liquid. After the mixing of the ingredients the composition should be kept at the high temperature for some minutes for cooking purposes and to ensure thickness.

Alternatively, the composition as disclosed above may be added to a cold aqueous liquid (e.g. water, wine, milk, stock, etc.), whereafter heat should be applied sufficiently to achieve a thickening effect.

Herbs are defined in The Illustrated Herbal Handbook by J. de Bañracli Levy, Faber and Faber Ltd., London, chapter 3. Spices are defined in "The Book of Spices", Livingstone Publ. Comp. 1969, p. 3.

The amount of herbs and/or spices preferably is 10 - 45 wt %, more preferably 20 - 30 wt %. The amount of nuts and/or seeds is preferably 10-20 wt %, more preferably 12-15 wt %.

The herbs and spices may be used as frozen herbs or spices, containing 10 - 95 wt % of water. Preferred herbs are selected from the group, consisting of sage, rosemary, basil, thymian, oregano, dill, parsley, garlic, onion, savory, majoram, dragon, red/green/yellow pepper and mixtures thereof known as Provençal and Italian, while the spices are selected from the group, consisting of black and white pepper, nut meg, mace, curry, curcuma, saffron, clove and mixtures thereof.

The edible salt can be derived from inorganic or organic acids or bases. The most preferred edible salt is NaCl. The preferred amount is 2 - 7 wt %.

5

The fat phase can contain small amounts of surfactants (up to 2 wt%). Suitable surfactants are e.g. mono/diglycerides-mixtures, but in general any known surfactant can be applied.

- 10 The fat blend is present in our compositions in an amount of 40 - 89 wt %, preferably 60 - 85 wt %, more preferably 70 - 80 wt %.

The liquid oil, which is applied in amounts of 90 - 99.8  
15 wt % in these fat blends can be selected from the group, consisting of: sunflower oil, high oleic sunflower oil, rapeseed oil, safflower oil, high oleic safflower oil, soyabean oil, coconut oil, maize oil, cotton seed oil, arachidic oil, olein-fractions of natural oils, such as  
20 palm oil olein, MCT - oils. The oil displays a solid fat content (NMR-pulse, not-stab.) of less than 10 % at 20 ° C. Preferably the amount of liquid oil is 93 - 99, most preferably 95 - 98 wt % in the fat blend.

- 25 The hard fat component of these blends must have the ability to form a crystal network in our compositions. This requirement is essential if stable compositions, wherein the herbs and/or spices are to remain dispersed during the shelf life of the compositions, are aimed for. Examples of  
30 suitable fats are hardened rapeseed oil, hardened sunflower seed oil, hardened soybean oil, hardened palm oil, hardened cottonseed oil or mixtures thereof. The best results were



obtained, by using hard fats, having an I.V.  $< 10$ , preferably containing  $> 40$  wt % of  $C_{20}$  - saturated fatty acids. A most preferred hard fat is fully hardened, high erucic rapeseed oil (Rp-70).

5

The pourable character of our compositions is guaranteed when the fat blend of liquid oil and 1-5 wt% hard fat-component has an apparent viscosity at 5 °C of 100-500 m.Pa.s. after storage for 5 days at 5 °C, at a shear of 200 s<sup>-1</sup>. The apparent viscosity at 20 °C being suitably between 50 and 300 m.Pa.s.

The apparent viscosity is measured with a Physica UM-MC-100 viscometer on a sample of about 20 ml of liquid oil, 15 containing 1-5 wt % of the hard fat-component. The blend is first stored for 5 days at measurement temperature, whereupon it is subjected to a shear of 200 s<sup>-1</sup>. The viscosity is measured after 2 min. shearing.

20 The flavour-component can be selected from etherical oils, natural or nature-identical flavour components or even artificial flavour compositions. Our compositions can be made by known techniques. Suitable procedures involve the preparation of a premix of liquid oil and hard fat, using 25 two A-units. After mixing of the oil and hard fat at 70-85 °C the mix can be transferred through an A1-unit, using an exit temperature of 0-10 °C and subsequently through an A2-unit, using a lower exit temperature of -8 to 5 °C. The ingredients can be added in one batch to the oil or can be 30 added in different batches to the fat mix during different phases of the process.

However we prefer to apply a process, wherein a cooling step is applied on the mixture of ingredients in the oil, prior to the addition of the hard fat component. In this way a better crystal network of the hard fat component can be established as a result of the addition of the hard fat component at a lower temperature. The thickener is usually mixed with part of the liquid oil which is optionally preheated. Therefore our invention also concerns a process for the preparation of our pourable fat-compositions, according to which process,

- (i) thickener is mixed with part of the liquid oil, which optionally is preheated
- (ii) the mix, resulting of (i) is optionally heated at 70 - 85 °C for 2 - 10 min.
- 15 (iii) the mix, resulting from (i) or the heated mix, resulting from (ii) is cooled to a temperature of maximum 25 °C
- (iv) the rest of the liquid oil is mixed with the hard-fat component at a temperature below  
20 30 °C
- (v) the products from (iii) and (iv) are mixed under the addition of the remaining components resulting in the product according to claims 1 - 9.

25

It was found, that the best ambient-stability was obtained, if in step (ii) the mix obtained in step (i) is heated to 70 - 85 °C for 2 - 10 min, preferably 4 - 8 min.

30 The mixing according to step (iv) can be performed by any conventional mixing technique. However it was found that

the best dispersions were obtained if this mixing was performed in a votator (= scraped surface heat-exchanger).

## 5 EXAMPLES

1. 280 g. of sunflower oil was heated to 80 °C. 100 g. of deepfrozen (- 20 °C) basil were added to this mix. The temperature was maintained at 75 °C for 5 min. by heating.  
10 The mixture obtained is cooled, while stirred, to 12 °C in about 10 min.

A mixture of 120 g. fat (composition: 92% sunflower oil and 8% hardened rapeseed oil m.pt. 70 °C) and 8 g. of Basil etheric oil (ex Tastemaker, art. no. KT 0117) as flavour  
15 ingredients and 24 g. NaCl were added to the mixture at 12 °C. An additional stirring during 10 min. was performed.

The product was collected.

20 2. The procedure of example 1 was repeated, however applying hardened sunflower oil m.pt. 69 °C in stead of hardened rapeseed oil m.pt. 70 °C. The amount of hardened sunflower oil was 4 wt% on total oil.

25 Above fat was applied for the preparation of compositions containing 44 wt% Dille or 44 wt% of a Provencal mix (=a mix of Marjoram herbs, Thyme herbs, Basil herbs, Rosemary herbs and Garlic paste in wt-ratio: 12.5: 2.5: 3.5: 2.5: 4.0).

30 The products obtained had acceptable properties, but were somewhat viscous and wither.

3. The procedure of example 2 was repeated, however using hardened rapeseed oil m.pt.70 °C in stead of hardened sunflower oil in an amount of 3 wt% on total oil.

5

The products obtained were not viscous and were not wither, so the products performed better than the products of example 2.

10 4. The procedure of example 3 was repeated, however applying a fat consisting of 25 wt% olive oil and 75 wt% sunflower oil.

The hardened rapeseed was used in an amount of 2.5 wt%.

15 Dille was applied as herb.

The product obtained displayed excellent properties.

5. Preparation of a liquid brown base sauce.

20

The following dry ingredients were mixed with the fat base and the liquid oil in the amounts indicated.

Ingredient	wt %
25 onion flavour	0.12
thickener-mixture	0.44
caramel powder	0.80
meat extract dried	1.16
30 milk protein	3.77
Col Flo (mod starch from NSC)	6.28
wheat flour	9.66

11

Gravy flavour (dried)	26.08
sunflower oil	3.40
pourable composition of example 3 without the herbs 48.3	

5 Mixing of 140 ml of this composition with 1 l of water resulted in the finished sauce.

6. Preparation of a white liquid base sauce.

10 The following dry ingredients were mixed with fat base and liquid oil.

Ingredient	amount in wt%
15 lactose	0.64 %
chicken flavour	0.99
Sauce Hollandaise dry flavour	1.71
skimmed milk powder	3.30
milk protein	3.64
20 instant wheat flour	3.64
coffee whitener	4.16
Col Flo (Nat starch)	6.60
Sauce flavours (dried)	11.19
sunflower oil	4.76
25 fat base from example 5	52.38

120 ml of this composition were mixed with 1 l full fat milk to provide the finished product.

## 7. Liquid Hollandaise base sauce

The following ingredients were mixed in the amounts indicated.

5		
	Ingredient	amount wt%
	butter flavour	0.13
	citric acid	0.16
10	wheat flour	0.21
	thickener mixture	1.29
	sugar	1.17
	full milk powder	2.64
	coffee whitener	2.76
15	Dried Hollandaise sauce flavour	7.43
	Col Flo (Nat Starch)	8.75
	egg yolk powder (stabilised)	4.13
	salt	1.98
	fat base of example 5	69.57
20		

## 8 Preparation of Liquid Roux

25 500 g of the fat base as made in example 5 were mixed with 500 g of Dalgety flour M 2200 (ie a physically modified wheat flour). The resulting product was used to bind stock to make a Veloute by mixing with the stock and bring back to boil and simmering shortly in dillution with. Dillution  
30 rate: 100 g sauce base in 1 liter stock.

## 9. Preparation of liquid binder

450 g of Col Flo (from Nat Starch Comp) were mixed with  
550 g of the fat base of example 5.

5 The resulting mixture was applied for the preparation of  
sauces after mixing it with hot water and bringing it back  
to the boil (1 part binder, 10 parts water).

## 10. Brown sauce

10

A mixture was prepared containing 50 wt% of the fat mixture  
as in Example 3 which was mixed with the following dry  
ingredients:

15 Ingredient	amount wt%
Dalgety flour M2250	23.5
Colflo 67 (National Starch)	4.4
Lygomme KH6 (gum mixture)	0.32
20 salt	3.1
Flavocuring composition	17.92
Bolec M (lecithine)	0.76
fat phase according to example 3	50.0

25

The resulting mixture was applied for the preparation of  
sauces after mixing it with hot water (dillution: 1 part  
concentrate, 6 parts water), bringing back to the boil and  
simmering for 3 minutes.

30

## 11. Curry sauce

A mixture was prepared containing 54 wt% of the fat mixture as in Example 3 which was mixed with the following dry ingredients:

5 Ingredient	amount wt%
Colflo 67 (National Starch)	10.0
wheat starch	15.0
Sugar	8.8
salt	3.0
10 Flavour composition	9.7
fat phase according to example 3	53.5

The resulting mixture was applied for the preparation of 15 sauces after mixing it with hot water (dillution: 1 part concentrate, 4 parts water), bringing back to the boil and simmering for 3 minutes.

## 20 12. White sauce

A mixture was prepared containing 60 wt% of the fat mixture as in Example 3 which was mixed with the following dry ingredients:

25 Ingredient	amount wt%
wheat starch	11.0
Colflo 67	11.0
creamer	10.0
salt	2.3
30 dried stabilised egg yolk	1.6
Flavour composition	2.5
Emulitop (lecithin)	1.6



fat phase according to example 3 60

The resulting mixture was applied for the preparation of 5 sauces after mixing it with hot water (dillution: 1 part concentrate, 5 parts water), bringing back to the boil and simmering for 3 minutes.

## Claims

1. Pourable fat-composition, that is ambient-stable, wherein the fat-composition comprises:
  - 0 - 45 wt % of herbs, spices, nuts and/or seeds
  - 5 - 60 wt % of a thickener, in particular selected from natural or modified starches, natural or modified gums and gelatins
  - 0 - 10 wt % of milk proteins
  - 0 - 10 wt % of egg yolk powder
  - 0 - 5 wt % of emulsifier
  - 0 - 35 wt % of meat-extract or gravy flavour
  - 1 - 10 wt % of an edible salt
  - 30- 89 wt % of a fat blend, comprising:
    - (i) 90 - 99.8 wt % of liquid oil
    - (ii) 10 - 0.2 wt % of a hard fat component, that has the ability to form a crystal network in the endproduct, preferably being hardened high erucic rapeseed oil,while the total-composition is flavoured by the presence of 0 - 20 wt % on total composition of flavour-ingredients.
2. Pourable fat-composition according to claim 1, wherein the herbs and/or spices are frozen fresh herbs and/or spices, containing 10 - 95 wt % of water.
3. Pourable fat-composition according to claims 1 or 2, wherein the herbs are selected from the group, consisting of sage, rosemary, basil, thymian, oregano, dill, parsley, garlic, onion, savory, majoram, dragon, red/green/yellow pepper and mixtures thereof known as

Provençal and Italian, while the spices are selected from the group, consisting of black and white pepper, nut meg, mace, curry, curcuma, saffron, clove and mixtures thereof.

4. Pourable fat-composition, according to claim 3, wherein the salt is NaCl.
5. Pourable fat-composition, according to claim 1, wherein the liquid oil is selected from the group, consisting of: sunflower oil, high oleic sunflower oil, safflower oil, high oleic safflower oil, soyabean oil, maize oil, cotton seed oil, arachidic oil, olein-fractions of natural oils, such as palm oil olein, MCT - oils.
6. Pourable fat-composition according to claim 5, wherein the oil displays a solid fat-content (NMR-pulse; not-stab.) of less than 10 % at 20 °C.
7. Pourable fat-composition according to claims 1 - 6, wherein the hard fat has an Iodine value of less than 10 and contains preferably more than 40 wt % of C<sub>20+</sub> - saturated fatty acids.
8. Pourable fat-composition according to claims 1 - 7, wherein the fat blend of liq. oil and 1-5 wt % hard fat-component has an apparent viscosity at 5 °C of 100-500 m.Pa.s. after storage for 5 days at 5 °C, or at 20 °C of 50-300 m.Pa.s. after storage for 5 days at 20 °C, both at a shear of 200 s<sup>-1</sup>.

9. Pourable fat-composition according to claim 1, wherein the flavour-component is selected from etherical oils, natural or nature-identical flavour components.
10. Process for preparing a pourable fat-composition according to claim 1-9, wherein:
  - (i) thickener is mixed with part of the liquid oil, which optionally is preheated
  - (ii) the mix, resulting of (i) is optionally heated at 70 - 85 °C for 2 - 10 min.
  - (iii) the mix, resulting from (i) or the heated mix, resulting from (ii) is cooled to a temperature of maximum 25 °C
  - (iv) the rest of the liquid oil is mixed with the hard-fat component at a temperature below 30 °C
  - (v) the products from (iii) and (iv) are mixed under the addition of the remaining components resulting in the product according to claims 1 - 9.

# INTERNATIONAL SEARCH REPORT

International Application No. <b>PCT/EP 98/02269</b>		
<b>A. CLASSIFICATION OF SUBJECT MATTER</b> IPC 6 A23D9/007 A23D9/04 A23L1/221 A23L1/36 A23L1/39		
According to International Patent Classification (IPC) or to both national classification and IPC		
<b>B. FIELDS SEARCHED</b> Minimum documentation searched (classification system followed by classification symbols) IPC 6 A23D A23L		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practical, search terms used)		
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>		
Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 0 536 976 A (BUNGE FOODS CORP) 14 April 1993 see page 3, line 33 - page 4, line 14 see claim 1	1-10
Y	WO 95 28849 A (US GOVERNMENT) 2 November 1995 see page 10, paragraph 3 see page 17, last paragraph - page 18, paragraph 1 see claims 1, 10-13, 18, 22	1-10
	-/-	
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Date of the actual completion of the international search <b>13 August 1998</b>		Date of mailing of the international search report <b>21/08/1998</b>
Name and mailing address of the ISA European Patent Office, P.O. 5918 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 851 epo nl Fax. (+31-70) 340-3018		Authorized officer <b>Dekeirel, M</b>

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International Application No.

PCT/EP 98/02269

## C. (Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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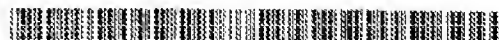
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(19) World Intellectual Property  
Organization  
International Bureau



(43) International Publication Date  
17 February 2005 (17.02.2005)

PCT

(10) International Publication Number  
**WO 2005/014158 A1**

(51) International Patent Classification<sup>7</sup>: **B01J 2/04**,  
A23D 7/00, 7/04

(21) International Application Number:  
PCT/EP2004/006544

(22) International Filing Date: 16 June 2004 (16.06.2004)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:  
03077247.9 17 July 2003 (17.07.2003) EP

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(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GR, GU, HD, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MY, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RU, RW, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, CH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— with international search report  
— before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments

For two-letter codes and other abbreviations, refer to the "Guideance Notes on Codes and Abbreviations" appearing in the beginning of each regular issue of the PCT Gazette.

(54) Title: PROCESS FOR THE PREPARATION OF AN EDIBLE DISPERSION COMPRISING OIL AND STRUCTURING AGENT

(57) Abstract: The invention relates to a process for the preparation of an edible dispersion comprising oil and structuring agent and one or more of an aqueous phase and/or a solid phase, in which the dispersion is formed by mixing oil, solid structuring agent particles and the aqueous phase and/or the solid phase, wherein the solid structuring agent particles have a microporous structure of submicron size particles.

WO 2005/014158 A1

Process for the preparation of an edible dispersion  
comprising oil and structuring agent

Description

5

Field of the invention

The present invention relates to a process for the preparation of an edible dispersion comprising oil and structuring agent, in particular to such dispersions  
10 comprising oil and structuring agent as continuous phase and a dispersed phase. The dispersed phase may be an aqueous liquid (thus forming a water-in-oil emulsion) or a solid particulate matter (thus forming a suspension). The invention further relates to the use of micronised fat  
15 powder to stabilise oil-containing dispersions.

Background of the invention

Edible dispersions comprising oil and structuring agent are well known. Examples of well-known products that  
20 substantially consist of such edible dispersions are water-in-oil emulsions, such as for instance margarines and spreads. These edible dispersions typically have an oil phase that is a blend of liquid oil and fat that is solid at normal ambient temperature (20°C). This solid fat, often  
25 also designated as hardstock, acts as structuring agent, and its function is to stabilise the dispersion. For a margarine or spread, ideally the structuring agent has such properties that it should have melted or dissolved at mouth  
30 mouthfeel.

Other known dispersions comprising oil and structuring agent are disclosed in EP-A-775444 and WO 98/47386. Herein the dispersed phase is a dry particulate matter, such as e.g. flour, starch, salt, spices, herbs etc.

5

Generally, the edible dispersions comprising structuring agent are prepared according to prior art processes that encompass the following steps:

- 1) dispersion of the aqueous phase and/or the solid phase  
10 through the oil phase, at a temperature where the oil phase, including the structuring agent is liquid;
- 2) formation of a fat crystal network to stabilise the resulting dispersion and give the product some degree of firmness;
- 15 3) modification of the crystal network to produce the desired firmness and confer plasticity.

These steps are usually conducted in a process that involves apparatus that allow heating, cooling and  
20 mechanical working of the ingredients, such as the churn process or the votator process. The churn process and the votator process are described in Ullmanns Encyclopedia, Fifth Edition, Volume A 16 pages 156-158. Using these techniques excellent dispersions (spreads) having high  
25 emulsion stability and good melting properties in the mouth can be prepared.

However, a disadvantage of the known processes is that the process involves a heating step and a cooling step and  
30 therefore requires a lot of energy. In a dispersion with for instance 4 wt.% structuring agent the whole weight of the dispersion (100 wt.%) needs to be heated and cooled.

Another disadvantage of the known processes is that the choice of fats that can practically be used as structuring agent is rather limited. If the melting point of the structuring agent is too high the melting properties in the mouth are unsatisfactory. If on the other hand, the melting point is too low, the emulsion stability will be negatively affected. Moreover the amount of saturated fatty acids in the structuring agent is usually relatively high. Saturated fatty acids are a known risk factor for cardiovascular health.

Further disadvantage of the known processes is that the product may deteriorate due to the changes in temperature caused by the heating and cooling step and that heat-sensitive ingredients cannot be incorporated.

Powdered fat is well known in the prior art. It may be prepared according to various processes, known in the art. Micronised fat is also known in the prior art. EP-B-744992 describes the preparation of micronised fat particles by dissolution of gas (carbondioxide) in the fat under pressure and decompressing the mixture in such way that the temperature falls below the solidification point of the fat, so that micronised particles are formed.

EP-A-1238589 describes a method for forming a food product, which contains an emulsion in which the food product in liquid form is contacted with a cryogen so as to cool the liquid product and effect a rapid conversion of the liquid to a solid. A disadvantage of this known process is that

still the whole emulsion has to be heated above the melting point of the structuring agent.

#### Summary of the invention

5 It is therefore an object of the invention to provide a process that requires less energy for the preparation of a dispersion comprising the structuring agent. Another object is to provide such a process that allows the use of more types of structuring agent, especially more sorts of  
10 hardstock. A further object of the invention is a reduction of the amount of saturated fatty acids in the hardstock. Still a further object of the invention is to provide a process for the preparation of a dispersion that allows the incorporation of heat-sensitive ingredients and/or that  
15 avoids deterioration of the emulsion.

One or more of these objects is attained according to the invention that provides a process for the preparation of an edible dispersion comprising oil and structuring agent and  
20 one or more of an aqueous phase and/or a solid phase, in which the dispersion is formed by mixing oil, solid structuring agent particles and the aqueous phase and/or the solid phase, wherein the solid structuring agent particles have a microporous structure of submicron size  
25 particles. Preferably, the solid structuring agent particles are at least 50% alpha-polymorph.

According to the invention the heating and cooling step of the emulsion ingredients that is needed in the prior art processes may be omitted or reduced and a stable dispersion  
30 can be made.

Preferably, the solid structuring agent particles are at 50% or more alpha-polymorph, more preferably 70% or more alpha-polymorph and most preferably 90% or more alpha-polymorph.

5

#### Detailed description of the invention

The invention relates to a process for the preparation of a dispersion. A dispersion is herein defined as a system in which two or more phases that are insoluble or only  
10 slightly soluble are distributed in one another.

The dispersion may be an emulsion, a suspension or foam or any combination thereof, it may be oil continuous, water continuous or bi-continuous. Preferably the dispersion is  
15 oil continuous, more preferably an oil continuous emulsion or oil continuous suspension.

Where a solid phase is present in the dispersion according to the invention, it is preferably a solid phase of dry  
20 particulate matter.

Where an aqueous phase is present in the dispersion according to the invention, it is preferably a dispersed aqueous phase.

25

According to the invention, the dispersion is formed by mixing oil, the solid structuring agent particles and the other phase or phases of the dispersion, such as for example an aqueous phase, a solid phase and/or a gas phase.  
30 The mixing of the ingredients may be done in any order, i.e. the ingredients/phases may all be mixed in one mixing step or alternatively the mixing may be executed in more

than one step. For instance an oil phase with the structuring agent particles may be mixed and a water phase may be prepared separately and later mixed with the oil phase.

5

According to the invention, the solid structuring agent particles should have a microporous structure of submicron size particles. An example of a microporous structure according to the invention is shown in figures 6 and 7  
10 hereafter. The submicron particles typically have the shape as shown in figure 7, and consist of platelets with submicron dimensions. The thickness of the platelets should be submicron, preferably the thickness is on average 0.01-0.5  $\mu\text{m}$ , more preferably 0.03-0.2  $\mu\text{m}$ , even more preferably  
15 0,06-0.12  $\mu\text{m}$ .

Equivalent good results were obtained for a microporous structure of more bubble-like shape, such as shown in  
20 figure 10, hereafter. In such microporous structure the wall thickness of the bubbles should be submicron, for instance on average 0.01-0.5  $\mu\text{m}$ , more preferably 0.03-0.2  $\mu\text{m}$ , even more preferably 0,06-0.12  $\mu\text{m}$ .

25

The microporous structure, may, in the course of the preparation of the dispersion, for instance through the force of a mixer, be broken into submicron particles. The resulting submicron particles will form the structuring  
30 network of the dispersion.

Preferably, the structuring agent is edible fat. Edible fats consist predominantly of triglycerides. Typically such

edible fats suitable as structuring agent are mixtures of triglycerides, some of which have a melting point higher than room or ambient temperature and therefore contain solids in the form of crystals.

5

The solid fat structuring agent, also denoted as hardstock or hardstock fat, serves to structure the fat phase and helps to stabilise the dispersion.

10 For imparting to common margarine a semi-solid, plastic, spreadable consistency this stabilising and structuring functionality plays an important role. The crystals of the solid fat form a network throughout the liquid oil resulting into a structured fat phase. The aqueous phase  
15 droplets are fixed within the spaces of the lattice of solid fat crystals. In this way coalescence of the droplets and separation of the heavier aqueous phase from the fat phase is prevented.

20 Generally, fats with a high content of HUH triglycerides show good structuring properties. H denotes a C16-C24 saturated fatty acid residue, such as palmitic acid (C16) or stearic acid (C18) and U denotes an unsaturated C18 fatty acid residue, such as oleic acid (C18:1) or linoleic  
25 acid (C18:2). Examples of suitable edible fat structuring agents (hardstock fats) are palm oil partially hydrogenated to a melting point of 44°C or an interesterified mixture of palm oil and a lauric fat.

30 Further common ingredients of the fat phase are emulsifiers, such as monoglycerides and lecithin, colouring agents and flavours.



The structuring agent should be added to the dispersion in the form of solid structuring agent particles. Preferably the solid structuring agent particles should have an alpha-5 polymorph.

The following nomenclature of the polymorphic forms of the structuring agent is used herein:

- 10 1.  $\alpha$ -polymorph (alpha polymorph): a form that gives only one short-spacing line in the X-ray diffraction pattern near 4.15 Å.
2.  $\beta'$ -polymorph (beta-prime polymorph): a form that gives  
15 two short spacing lines near 3.80 Å and 4.20 Å in the X-ray diffraction pattern and also shows a doublet in the 720  $\text{cm}^{-1}$  in the infrared absorption spectrum
3.  $\beta$ -polymorph (beta polymorph): a form that does not  
20 satisfy criteria 1. or 2.

See for an explanation of polymorphism and the above definition: Gunstone, F.D.; Harwood, J.L.; Padley, F.B.; The Lipid Handbook, second edition, Chapman and Hall, page  
25 405.

The solid structuring agent particles preferably have an average particle size ( $D_{3,2}$ ) of 60 micrometer or less, more preferably the solid structuring agent particles have an  
30 average particle size of 30 micrometer or less. The average particle size ( $D_{3,2}$ ) is determined as indicated in the examples.

Preferably the solid structuring agent particles are prepared using a micronisation process. In the micronisation process the solid structuring agent particles  
5 are prepared by preparing a homogeneous mixture of structuring agent and liquified gas or supercritical gas at a pressure of 5-40 MPa and expanding the mixture through an orifice, under such conditions that a spray jet is applied in which the structuring agent is solidified and  
10 micronised. The liquified gas or supercritical gas may be any gas that may be used in the preparation of food products, for example carbondioxide, propane, ethane, xenon or other noble gases. Carbondioxide and propane are preferred. Carbondioxide is most preferred. Advantages of  
15 carbondioxide are that it has a mild (31°C) critical temperature, it is non-flammable, nontoxic, environmentally friendly and it may be obtained from existing industrial processes without further contribution to the greenhouse effect. It is fairly miscible with oil and is readily  
20 recovered owing to its high volatility at ambient conditions. Finally liquid CO<sub>2</sub> is the second least expensive solvent after water.

The temperature of the mixture of structuring agent and  
25 liquified gas or supercritical gas is preferably such that the mixture forms a homogeneous mixture. Advantageously, the temperature of the mixture of structuring agent and liquified gas or supercritical gas is below the slip melting point of the structuring agent at atmospheric  
30 pressure and above the temperature at which phase separation of the mixture occurs. Under such conditions the smallest micronised particles may be obtained.

The pressure and temperature of the mixture of structuring agent and liquified or supercritical gas is preferably such that a large amount of the gas may be dissolved in the structuring agent. The amount dissolved will be determined by the phase diagram of the mixture of structuring agent and liquified or supercritical gas. At higher pressures as well as at lower temperatures more gas will dissolve in the structuring agent.

10

Preferably the temperature and pressure are chosen such that 10 wt.% or more, more preferably 20 wt.% or more or most preferably 30 wt.% or more of gas is dissolved in the liquid phase. The mixture of structuring agent and liquified or supercritical gas may contain additional substances, such as for instance oil. We have found that the addition of oil may reduce sintering of the micronised particles of the structuring agent.

20 The mixture containing structuring agent and liquefied or supercritical gas is depressurised over a small orifice or nozzle, to break up the mixture into small droplets. The break-up of the mixture into droplets can be assisted e.g. by internals inside the nozzle before the orifice to generate a whirl, or by passing a gas at a high flow rate near the orifice.

The mixture is depressurised into a volume where the pressure is higher than, equal to or lower than atmospheric pressure.

We have found that sintering, agglomeration and ripening of micronised particles of the structuring agent will lead to

a reduced performance of the particles for structuring the dispersion.

To avoid sintering, agglomeration and/or ripening of the 5 micronised particles, preferably a gas jet is applied in addition to the flow of the spray jet. The additional gas jet is most effective when the gas jet is positioned such that recirculation of material expanded through the orifice is reduced or avoided. Especially advantageous is a 10 position wherein the gas from the gas jet flows essentially tangentially to the flow direction of the spray jet. Most advantageously the gas inlet for the gas jet is positioned behind the exit of the nozzle, see figure 2. Figure 2 shows that the additional gas inlet (1) behind the exit of the 15 nozzle (2) creates a gas flow (3) tangentially to the flow of the spray jet (4).

To further avoid agglomeration and ripening, the spray jet is preferably sprayed into a collection chamber, and a flow 20 of gas having a temperature lower than the slip melting point of the structuring agent is fed into the collection chamber.

Preferably the edible dispersion according to the invention 25 is a water and oil containing emulsion, optionally including a solid phase. The emulsions are preferably oil continuous. Examples of suitable emulsions are table spreads, dressings, soups, sauces, shortenings, cooking oils, frying oils, whipping creams and mayonnaises.

30

A stable dispersion is herein defined as dispersion that shows an oil exudation of less than 5% after storage for 15

weeks at 15°C, measured according to the method described in the examples.

- A further preferred edible dispersion according to the invention is a dispersion of a solid matter, preferably a dry particulate matter, dispersed in a continuous phase of oil and structuring agent. Preferred material for the dry particulate matter is one or more of flour, starch, salt, herbs (e.g. dried herbs), spices and mixtures thereof.
- 10 Preferably in such dispersions, the amount of solid matter is 30-75 wt.%, more preferably 40-65 wt.% based on total weight of the dispersion.

- The amount of structuring agent should be such that a suitably stable dispersion is obtained. When the structuring agent is micronised fat, the amount is preferably 1-20 wt.%, more preferably 4-12 wt.% based on total weight of the dispersion.

## 20 Description of the figures

- Figure 1: Schematic view of the micronisation apparatus used in the examples
- 25 Figure 2: Schematic view of the nozzle configuration with gas inlet for tangential gas-flow.
- Figure 3: SEM Photograph of micronised fat powder prepared in example 1 (magnification 250x)

- Figure 4: SEM Photograph of micronised fat powder  
prepared in comparative experiment A  
(magnification 250x)
- 5 Figure 5: SEM Photograph of micronised fat powder  
prepared in comparative experiment B  
(magnification 250x)
- Figure 6: SEM Photograph of micronised fat powder  
10 prepared in example 1 (magnification 1000x)
- Figure 7: Enlarged SEM photograph of the micronised  
fat powder of example 1
- 15 Figure 8: Enlarged SEM photograph of the micronised  
fat powder of example 8
- Figure 9: Enlarged SEM photograph of the micronised  
fat powder of example 9
- 20 Figure 10: Enlarged SEM photograph of the micronised  
fat powder of example 10

25

Examples

### General

#### Method to determine slip melting point

The slip melting point of structuring agent is determined in accordance with F. Gunstone et al, The Lipid Handbook, second edition, Chapman and Hall, 1995, page 321, Point 5.2.3, Slip point.

#### Method to determine $D_{3,2}$ of the particle size distribution of micronised fat particles

Low-angle laser light scattering (LALLS, Helos Sympatic) was used to measure the average particle size ( $D_{3,2}$ ). The fat particles were suspended in water in a quixel flow cuvette with an obscuration factor of 10-20%. The diffraction pattern was measured at 632.8 nm with a lens focus of 100 mm and a measurement range of 0.5-175  $\mu$ m. Calculations were based on the Fraunhofer theory.

A full description of the principle of LALLS is given in ISO 13320-1.

#### Method to determine $D_{3,2}$ of water droplet size distribution in an emulsion

The water droplet size was measured using a well-known low resolution NMR measurement method. Reference is made to Van den Enden, J.C., Waddington, D., Van Aalst, H., Van Kralingen, C.G., and Packer, K.J., Journal of Colloid and Interface Science 140 (1990) p. 105.

Method to determine oil exudation

Oil exudation is determined by measuring the height of the free oil layer that appears on top of the product. This free oil layer is considered a product defect. In order to  
5 measure oil exudation, the product is filled into a scaled glass cylinder of 50 ml. The filling height is 185 mm. The filled cylinder is stored in a cabinet at constant temperature (15°C). Height measurements are executed every week, by measuring the height of the exuded oil layer in mm  
10 with a ruler. Oil exudation is expressed as the height of the exuded oil layer divided by the original filling height and expressed in %. Shaking of the cylinders should be avoided.

Method to determine pourability

15 Pourability for pourable compositions according to the invention is measured according to the standard Bostwick protocol. The Bostwick equipment consists of a 125 ml reservoir provided with a outlet near the bottom of a horizontally placed rectangular tub and closed with a  
20 vertical barrier. The tub's bottom is provided with a 25 cm measuring scale, extending from the outlet of the reservoir. When equipment and sample both have a temperature of 15°C, the reservoir is filled halfway with 62.5 ml of the sample after it has been shaken by hand ten  
25 times up and down. When the closure of the reservoir is removed the sample flows from the reservoir and spreads over the tub bottom. The path length of the flow is measured after 15 seconds. The value, expressed as cm per 15 seconds is the Bostwick rating, which is used as  
30 yardstick for pourability.



### Example 1

#### *Fat micronisation*

5

A set-up was constructed to dissolve carbon dioxide in the melt and expand the mixture over a nozzle to atmospheric pressure. The micronised product was collected in a drum (6) of 250 liters. The set-up is illustrated in figures 1.

10

**Autoclave** The equipment consists of a 1-liter autoclave (2) equipped with a mechanical stirrer (6-blade turbine impeller), a water jacket for heating and a Pt-100 resistance thermometer. The inner diameter of the autoclave is 76 mm. The autoclave has connections at the top and at the bottom.

15

**Tubing** The bottom connection of the vessel was used to pressurise the system with carbon dioxide or to lead the mixture to the nozzle. A 3-way valve (12) is used to switch between CO<sub>2</sub> supply (1) and nozzle (3). To expel the mixture from the vessel the CO<sub>2</sub> is supplied to the top of the autoclave via valve (11). The length of tube between the bottom connection and the nozzle (3) is approximately 30 cm. All tubing has an outer diameter of 1/4" (inner diameter approximately 1/8") and is equipped with electrical tracing. Additional gas, N<sub>2</sub> or He, can be supplied through (10) to maintain a

20

25

30

constant pressure inside the autoclave during the expansion over the nozzle

- Nozzle
- 5 The nozzle (3) can be designed with different orifice diameters (opening outlet) and cores (construction of the supply to the orifice). For this work nozzles were used with an orifice of 0.34 mm and standard core. The nozzle was heated by electrical tracing and
- 10 its temperature was registered by a thermocouple Pt-100.
- Collection
- 15 The nozzle was mounted to a Perspex tube (7) of 30 cm diameter and 20 cm length to allow observation of the jet during expansion. This transparent Perspex tube (7) with the nozzle (3) was mounted on top of an oil-drum (6) (250 liters) with a removable lid, which served as the collection chamber. The lid of
- 20 the drum has an outlet (8) to allow the expanded CO<sub>2</sub> to escape. A separator (9) retains the solid particles in the collection chamber. An additional gas jet (CO<sub>2</sub>) may be supplied through nozzle (4) connected to a gas
- 25 supply (CO<sub>2</sub> bottle) (5).
- Loading
- 30 The equipment was heated to the required temperature. Approximately 300 grams of fat (RP70, rapessed oil hardened to a slip melting point of 70° C) was completely melted and heated to 20 degrees above its melting point and charged into the autoclave.

- Equilibrium      The autoclave was pressurised in about 10 minutes through the bottom connection. During pressurisation the CO<sub>2</sub> supply to the top was closed. After reaching the final pressure the top valve was opened and the 3-way valve was closed. The melt was allowed to absorb CO<sub>2</sub> and equilibrate for 30 minutes, while stirring the mixture and supplying additional CO<sub>2</sub>. The equilibrium pressure in the autoclave was 15 MPa and the temperature in the autoclave was 60°C.
- Expansion      To expand the melt the stirrer was stopped and the supply of additional gas to the collection chamber was turned on. Next the 3-way valve was switched to supply the mixture to the nozzle. During expansion of the mixture in example 1 the pressure in the autoclave was maintained by the CO<sub>2</sub> supply. In examples 2 and 3 the pressure in the autoclave was increased to and maintained at 15 MPa by supplying He to the top of the vessel, after first equilibrating with CO<sub>2</sub>.
- A micronised fat powder that was obtained which was a very fine and dry solid powder. The powder was 100% alpha-polymorph. In the X-ray diffractogramme, peaks for the  $\beta'$  and  $\beta$ -polymorph were totally absent. The micronised fat powder was stored at 5°C. When stored at

5°C the micronised fat powder stayed 100%  
alpha-polymorph during more than one month.

The micronisation parameters are given in table 2.

5

Preparation of an edible water-in-oil emulsion

A pourable margarine was prepared with the composition  
shown in table 1:

10

Table 1: Composition of pourable margarine

Ingredient	Amount (wt.%)
<i>Oil phase</i>	
Sunflower oil	79.62
Micronised Rp 70 powder	1.95
Lecithin Bolec MT <sup>1</sup>	0.18
Fractionated lecithin Cetiol <sup>2</sup>	0.10
Beta-carotene (0.4wt.% solution in sunflower oil)	0.15
<i>Water phase</i>	
Water	16.5
Sodium chloride	1.5

Explanation of table 1:

15 The balance of all composition to 100% is water

RP 70 : Rapeseed oil hardened to a slip melting point of  
70 °C.

- 1: Lecithin was hydrolysed soybean lecithin (Solec MT) obtained from UMZ (Unimills Zwijndrecht, Netherlands)
- 2: Alcohol-soluble fraction from fractionation of native soybean lecithin with alcohol; Cetinol from UMZ.

5

The water phase was prepared by adding salt to distilled water and adjusting the pH of distilled water from 7.7 to 4.0 using 5 wt.% citric acid, and heated for 5 minutes in a bath of 60°C to dissolve the solids. The oil phase was prepared by dissolving the emulsifier ingredients and  $\beta$ -carotene in the total amount of sunflower oil at 15°C. Subsequently the micronised fat powder was added to the oil phase carefully using a spatula and the oil phase was mixed with a Turrax at 22000 rotations per minute (rpm) for 6 minutes. Then the water phase was added to the oil phase and the resulting mixture was mixed with a Turrax for 5 minutes at 23500 rpm in a water bath at having a temperature of 15°C.

20 The temperature of the mixture in the Turrax increased due to the viscous dissipation. However during the whole experiment the temperature was kept below 20°C The Turrax (type T50) was delivered by Janke & Kunkel IKA Labortechnik. This type of Turrax is designed to minimise 25 air entrainment.

The emulsion was partly poured into a glass cylinder and partly into a twist off pot of 100 ml and both were containers were stored in a cabinet at 15°C.

30

Results

The prepared emulsions were tested in accordance with the test methods described herein and the results of the tests are given in table 3. A SEM photograph of the micronised fat powder of example 1 (magnification 250 times) is given 5 in figure 3, with magnification of 1000 times in figure 6, and with magnification of 2000 times in figure 7.

#### Comparative experiment A

10 Comparative experiment A was conducted as example 1, however the fat micronisation step was modified in that the equilibrium pressure in the autoclave was 5 MPa instead of 15 MPa. Before and during depressurisation over the nozzle the mixture in the autoclave was pressurised with Helium to 15 15 MPa.

The results are shown in table 3. A SEM-photograph of the micronised fat powder is given in figure 4.

#### 20 Comparative experiment B

Comparative experiment B was conducted as example 1, however the fat micronisation step was modified in that the equilibrium pressure in the autoclave was 10 MPa instead of 25 15 MPa. Before and during depressurisation over the nozzle the mixture in the autoclave was pressurised with Helium to 15 MPa.

The results are shown in table 2. A SEM-photograph of the 30 micronised fat powder is given in figure 5.

All powders of example 1 and comparative experiments A and B showed the presence of 100% alpha-polymorph material. The micronised powder according to example 1 has a low particle size (see table 2) and has a microporous structure of submicron size particles, as is shown in figure 6. In contrast the powders of comparative experiments A and B have a higher particle size and a structure in which submicron size particles are not apparent.

10

Table 2: Micronisation parameters of example 1 and comparative experiments A and B

Example	Equilibrium Pressure (MPa)	Temperature (°C)	Amount of CO <sub>2</sub> dissolved (wt.%)	D <sub>3,2</sub> (µm)
1	150	60	19	39
A	50	70	7	72
B	100	60	16	75

Table 3: Oil exudation (%) of the emulsions of example 1 and comparative experiments A and B as function of the storage time at 15°C.

5

Storage time	Example 1	Comp. Ex. A	Comp. Ex. B
1 day			35.1
2 days			40.5
3 days	0		48.6
1 week	0	1.1	59.5
2 weeks	0	16.2	59.5
3 weeks	0	18.9	62.2
4 weeks			62.2
5 weeks			
6 weeks			
7 weeks	0.5	18.9	
8 weeks			
9 weeks			64.9
10 weeks			
11 weeks	0.5	18.9	
12 weeks			
14 weeks			64.9
15 weeks	0.5		
16 weeks		21.6	

The results show that the emulsion according to example 1 shows a very low oil exudation, which whereas those of comparative experiments A and B have a high oil exudation 10 and therefore the emulsions are not stable.



## Examples 2-4

5 Example 1 was repeated, but now instead of fat a mixture of  
fat and sunflower oil was micronised. The composition of  
the mixture of fat and oil is shown in table 3. In the  
preparation of the emulsion a Turrax speed of 8000 rpm was  
used and the Turrax time was 4 minutes.

10

Table 4: Micronisation parameters and emulsion properties  
of examples 2-4

Example	Fraction sunflower oil (wt.%)	Texture of micronised product	Bostwick (cm)	D <sub>0.3</sub> (µm)
2	22	Fine dry powder	14	4.36
3	50	Slightly granular somewhat sticky powder	14.6	3.06
4	75	Ointment like structure	10	-

15

All micronised products of examples 2-4 showed the presence  
of alpha-polymorph material in an amount of 100% and  
comprised submicron size particles. '-' means not

determined. Table 5: Oil exudation (%) of the emulsions of examples 2 to 4 as function of the storage time at 15°C.

Storage time	Example 2	Example 3	Example 4
1 day	5	0	0
4 days	18	0	0
5 days	40	0	0
1 week	45	0	0
2 weeks	52	0.5	0
3 weeks	52	0.5	0
4 weeks	52	1	0
6 weeks	52	1.5	0
8 weeks	55	2	0
10 weeks	55	2	0
12 weeks	55	2	0
14 weeks	55	2	0.5
16 weeks	55	2	0.5

5

Examples 2-4 show that the addition of oil to the structuring agent prior to micronisation leads to a reduction in oil exudation of the emulsion prepared using the micronised structuring agent. The micronised mixtures have a different appearance depending on the amount of oil added.

#### Example 5

15 Micronised fat was prepared according to example 1, fat micronisation using instead as fat rapeseed oil hardened to a slip melting point of 68°C.

A dispersion of solid matter in a fat phase was prepared by first preparing a mixture of 4.6 parts (all parts are weight parts) micronised fat in 4.6 parts sunflower oil and 5 stirring the mixture for 3 minutes at about 18°C under vacuum. The obtained mixture was added to 49 parts sunflower oil and mixed under vacuum at about 18°C for 1 minute.

10 To this mixture was added 41.2 parts flour and 0.6 parts parsley flakes (dried) and the resulting mixture was stirred under vacuum at about 18°C for 1 minute, 30 seconds. The resulting dispersion was stable for more than one month at room temperature without substantial oil 15 exudation.

#### Example 6

A dispersion was prepared with the following composition 20 (wt.% on final product):

Flour	49%
Dried herb pieces	1%
Sunflower oil	45%
25 Micronised fat powder (see example 5)	5%

The product was prepared by mixing all ingredients at room temperature using an ultraturrax mixing equipment. The product showed no oil exudation for one month.

### Example 7

A dispersion was prepared similar to that of example 6, however using 47.5 wt.% sunflower oil and 2.5 wt.% micronised fat prepared in example 1. The processing was the same. When stored at 5°C for one month, the product showed minimal oil exudation.

### Examples 8 to 10

10

Example 1 was repeated, however instead of Rp70, SF69 (sunflower oil hardened to a slip melting point of 69°C) was micronised and used as hardstock in the preparation of the emulsion.

15

To investigate how  $T_a$  (Equilibrium autoclave temperature) influences the morphology of the powders after micronisation, three different experiments were performed at  $T_a = T_m - 10^\circ\text{C}$  (Example 8),  $T_a = T_m - 5^\circ\text{C}$  (Example 9) and  $T_a = T_m$  (Example 10) respectively, with  $P = 180$  bar, in which  $T_m$  is the melting point of the hardstock, for Rp69 in these example 69°C.

Xray diffraction showed that all micronised powders are in the  $\alpha$  polymorph. SEM analysis shows no real differences in morphology within the chosen range of temperatures, although for  $T_m - 10^\circ\text{C}$  (59 °C) and  $T_m - 5^\circ\text{C}$  (64 °C) the morphology seems to be a little more brittle than for  $T_m$  (69 °C).

30

*Model emulsions*

Model emulsions were prepared using standard conditions and stored at 15 °C and 25 °C. In table 6, a summary of the measured oil exudation (O.E.) and Bostwick values (BW) as function of storage time is given.

5

Table 6: Results of Examples 8-10, Oil exudation (O.E. [%]) and Bostwick values (BW [cm]) as function of storage time and temperature

Example	Tm	P	Bostwick value [cm]			
	[°C]	[MPa]				
			Start	2wks	5wks	9wks
8	59	18	10	10	10	9
9	64	18	12	11	11	10
10	69	18	10	9	10	10
	O.E. at 15 °C			O.E. at 25 °C		
	2wks	5wks	9wks	2wks	5wks	9wks
8	0	0	0	0.8	1.1	1.5
9	0	0	0	0	1.1	1.5
10	0	0	0	1.5	3.8	5.3

10

Results show that at Tm of 59 °C and 64 °C, good O.E. and BW values after 9 weeks were achieved. At Tm=69 °C the oil exudation at 25° C is less favourable.

15

Enlarged SEM photographs (5000 x magnification) of the micronised powders of examples 8, 9 and 10 are shown in figures 8, 9 and 10 respectively.

## Claims

1. Process for the preparation of an edible dispersion comprising oil and structuring agent and one or more of an aqueous phase and/or a solid phase, in which the dispersion is formed by mixing oil, solid structuring agent particles and the aqueous phase and/or the solid phase, wherein the solid structuring agent particles have a microporous structure of submicron size particles.
2. Process according to claim 1, wherein the solid structuring agent particles are at least 50% alpha-polymorph.
3. Process according to claim 1 or 2, wherein the structuring agent is edible fat.
4. Process according to any of claims 1 to 3, wherein the edible dispersion is a water and oil containing emulsion, optionally including a solid phase.
5. Process according to claim 3 or 4, wherein the solid structuring agent particles have an average diameter  $D_{3,2}$  of 60  $\mu\text{m}$  or lower.
6. Process according to any of claims 1 to 5, wherein the solid structuring agent particles have an average particle size  $D_{3,2}$  of 30  $\mu\text{m}$  or lower.

7. Process according to any one of claims 1 to 6, wherein the solid structuring agent particles are prepared using a micronisation process.
8. Process according to any one of claims 1 to 7, wherein the solid structuring agent particles are prepared by preparing a homogeneous mixture of structuring agent and liquified gas or supercritical gas at a pressure of 5-40 MPa and expanding the mixture through an orifice, under such conditions that a spray jet is applied in which the structuring agent is solidified and micronised.
9. Process according to claim 8, wherein the homogenised mixture comprises oil.
10. Process according to claim 9, wherein the homogenised mixture comprises 10-90 wt.% based on the weight of the sum of oil and structuring agent.
11. Process according to claim 9 or 10, wherein temperature of the mixture of structuring agent and liquified gas or supercritical gas is such that the mixture forms a homogeneous mixture.
12. Process according to claim 11, wherein the temperature of the mixture of structuring agent and liquified gas or supercritical gas is below the slip melting point of the structuring agent at atmospheric pressure and above the temperature at which phase separation of the mixture occurs.

13. Process according to any of claims 8 to 12, wherein a gas jet is applied in addition to the spray jet.
14. Process according to claim 13, wherein the gas jet is positioned such that re-circulation of material expanded through the orifice is reduced or avoided.
15. Process according to the claim 13 or 14, wherein the gas from the gas jet flows essentially tangentially to the flow direction of the spray jet.
16. Process according to any of claims 8 to 15, wherein the spray jet is sprayed into a collection chamber, and a flow of gas having a temperature lower than the slip melting point of the structuring agent is fed into the collection chamber.
17. Use of micronised fat powder to stabilise an edible dispersion comprising oil.
18. Use according to claim 17, wherein the edible dispersion comprising oil is a water and oil containing emulsion, optionally including a solid phase.
19. Use according to claim 17, wherein the edible dispersion is a dispersion of 30-75 wt.% solid matter in oil.
20. Use according to claim 19, wherein the solid matter comprises dry particulate matter.



21. Use according to claim 20, wherein the dry particulate matter comprises one or more of flour, starch, salt, dried herbs, spices and mixtures thereof.

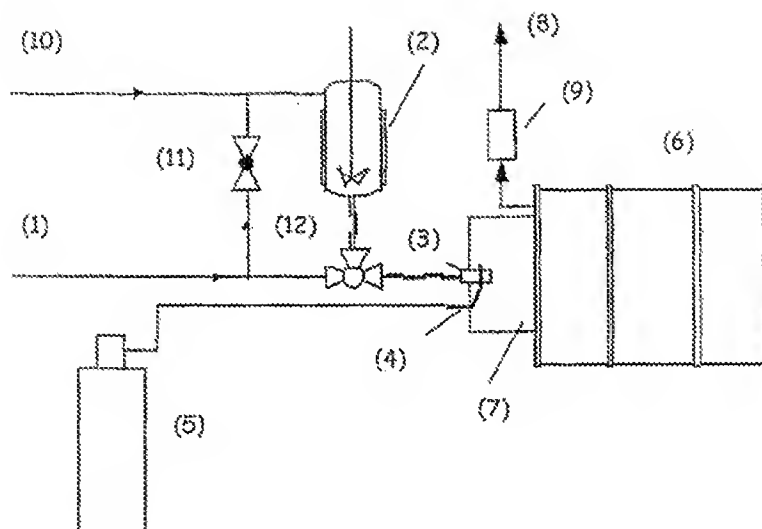


Fig. 1/10

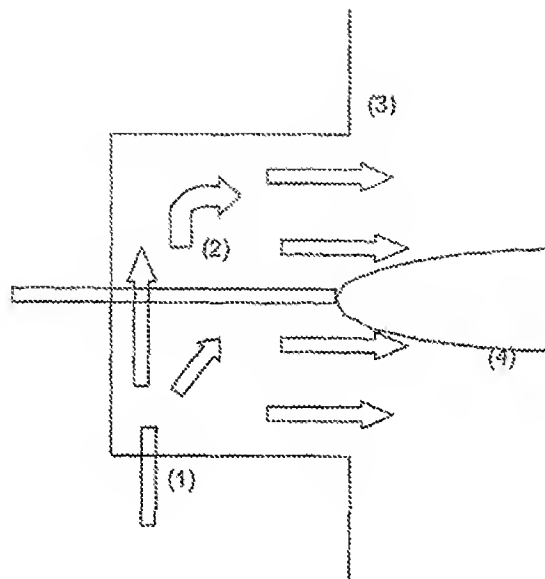


Fig. 2/10

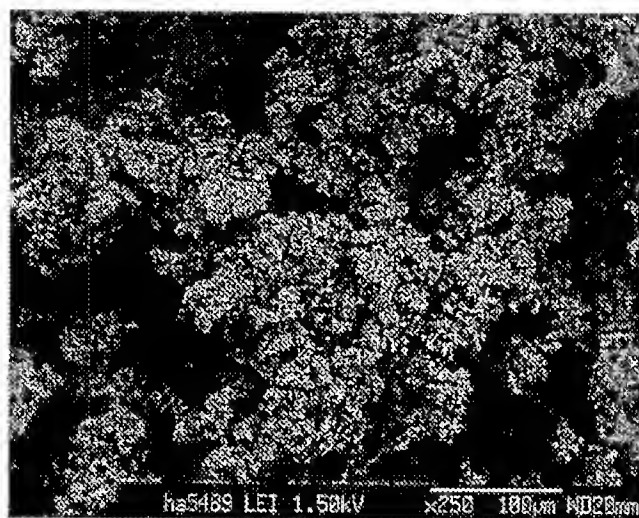


Fig. 3/10

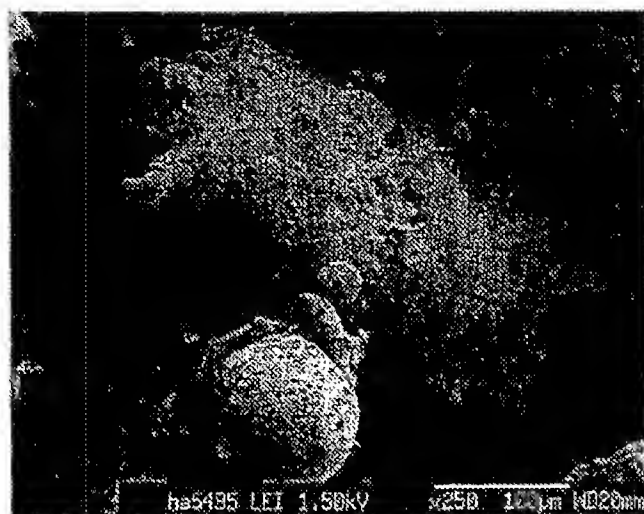


Fig. 4/10

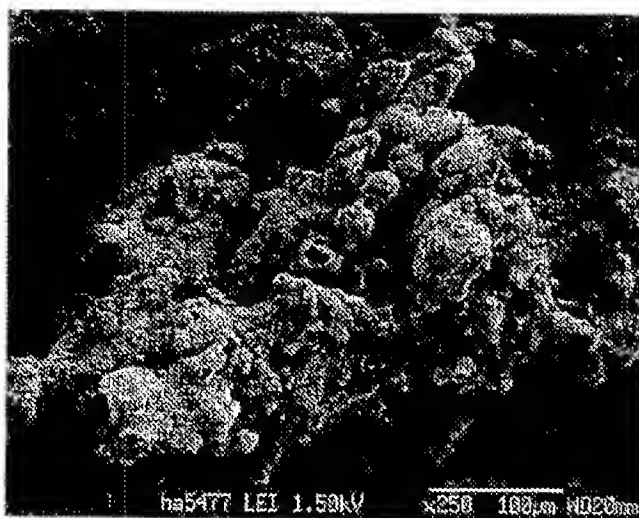


Fig. 5/10

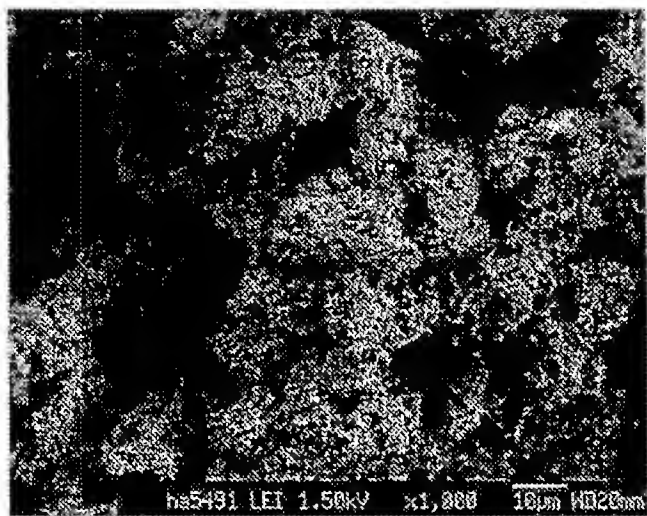


Fig. 6/10

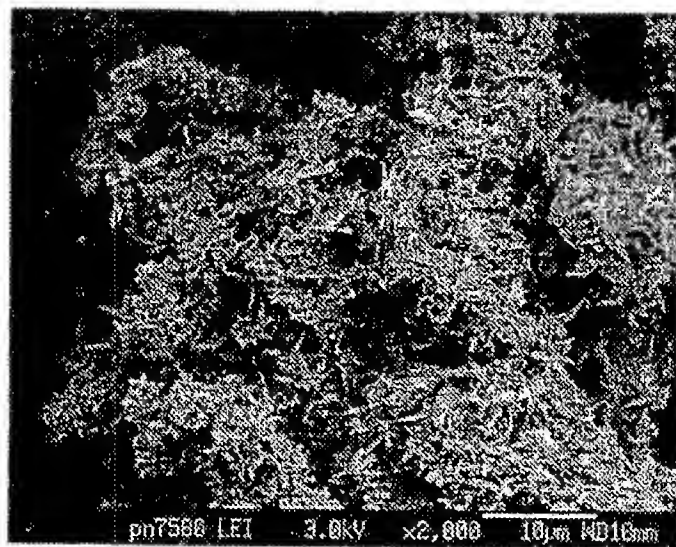


Fig. 7/10



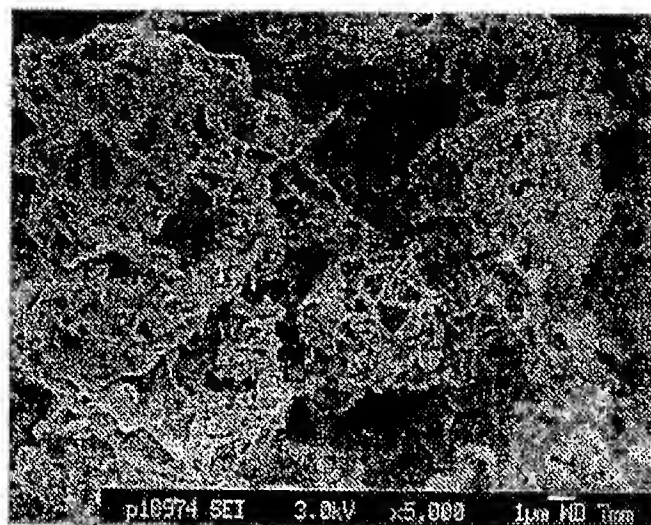


Fig. 8/10

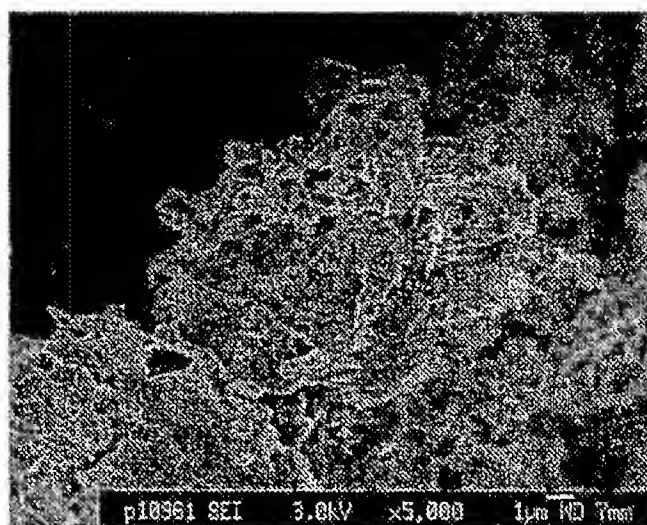


Fig. 9/10

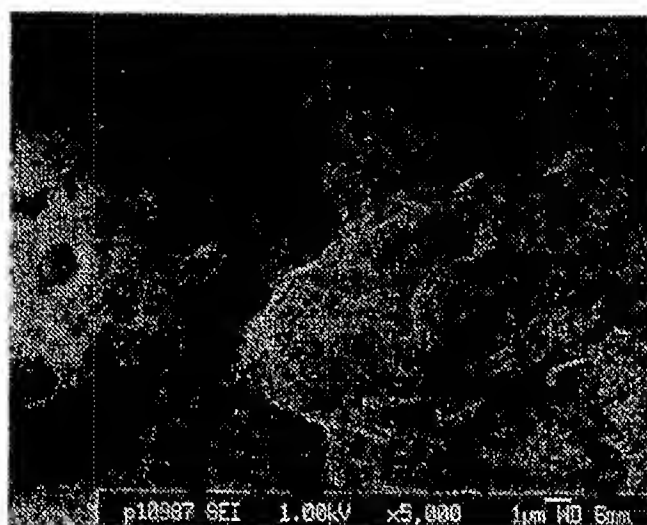


Fig. 10/10

# INTERNATIONAL SEARCH REPORT

International Application No.

PCT/EP2004/006544

<b>A. CLASSIFICATION OF SUBJECT MATTER</b> IPC 7 501J2/04 A2307/00 A2307/04		
According to International Patent Classification (IPC) or to both national classification and IPC		
<b>B. FIELDS SEARCHED</b> Minimum documentation searched (classification system followed by classification symbols) IPC 7 501J A23D		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practical, search terms used) EPO-Internal, WPI Data, FSTA, MEDLINE, BIOSIS		
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>		
Category	Description of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2002/048606 A1 (ZAWISTOWSKI JERZY) 25 April 2002 (2002-04-25) paragraphs [0007] - [0010], [0012] - [0031] paragraphs [0036] - [0041] paragraphs [0043] - [0051] examples 1,2	1,4-7
X	BELITZ H.D., GROSCH W.: "Food Chemistry, Second Edition" 1999, SPRINGER-VERLAG, BERLIN HEIDELBERG NEW YORK, XP002264854 ISBN: 3-540-64692-2 page 472; table 10.4	1,4-6
<div style="display: flex; justify-content: space-between;"> <span><input checked="" type="checkbox"/> Further documents are listed in the continuation of box C</span> <span><input checked="" type="checkbox"/> Patent family members are listed in annex</span> </div>		
<b>* Special categories of cited documents</b>		
<div style="display: flex;"> <div style="flex: 1;"> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="flex: 1;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"Z" document member of the same patent family</p> </div> </div>		
Date of the actual completion of the international search		Date of making of the international search report
5 November 2004		28.01.2005
Name and mailing address of the ISA European Patent Office, P.B. 5618 Patentlaan 2 NL - 2280 HV Rijswijk Tel: (+31-70) 340-2040, Tx: 31 051 epo nl Fax: (+31-70) 340-2010		Authorized officer:  Popa, M

## INTERNATIONAL SEARCH REPORT

International Application No.

PCT/EP2004/006544

## C. (Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	ANONYMOUS: "Particle sizes of milk powders" DAIRY INGREDIENTS FAX, vol. 2, no. 4, 8 August 2001 (2001-06-08), XP002264853 tables	1,4-6
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# INTERNATIONAL SEARCH REPORT

International Application No.

PCT/EP2004/006544

## C (Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Description of document, with indication, where appropriate, of the relevant passages	Relevant to exam. No.
A	US 2 521 219 A (HOLMAN GEORGE W ET AL) 5 September 1950 (1950-09-05) the whole document.	
A	----- TÜRK M ET AL: "Micronization of pharmaceutical substances by the Rapid Expansion of Supercritical Solutions (RESS): a promising method to improve bioavailability of poorly soluble pharmaceutical agents" JOURNAL OF SUPERCRITICAL FLUIDS, PRA PRESS, US, vol. 22, no. 1, January 2002 (2002-01), pages 75-84, XP004313515 ISSN: 0896-8446 the whole document -----	7-16

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/EP2004/006544

## Box II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:  
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☒ Claims Nos.:  
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:  
see FURTHER INFORMATION sheet PCT/ISA/210
3. ☐ Claims Nos.:  
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(b).

## Box III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this International application, as follows:

see additional sheet

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☒ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

1-16

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

## FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box II.2

Present claim 2, 5 and 6 relate to a product defined (inter alia) by reference to the following parameters:

- P1: ... the solid ... particles are at least 50% alpha-polymorph
- P2: ... an average diameter D3,2 of ...

The use of these parameters in the present context is considered to lead to a lack of clarity within the meaning of Article 6 PCT. It is impossible to compare the parameters the applicant has chosen to employ with what is set out in the prior art. The lack of clarity is such as to render a meaningful complete search impossible. Consequently, the search has been restricted to the rest of the features present in the claims.

The applicant's attention is drawn to the fact that claims relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure. If the application proceeds into the regional phase before the EPO, the applicant is reminded that a search may be carried out during examination before the EPO (see EPO Guideline C-VI, 8.5), should the problems which led to the Article 17(2) declaration be overcome.



FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. claims: 1-16

Process for obtaining an edible dispersion comprising oil.

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2. claims: 17-21

Use of micronised fat powder to stabilise an edible dispersion comprising oil.

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No.

PCT/EP2004/006544

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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